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LUFBERY AVENUE, WALLINGFORD, CONNECTICUT

Technical Progress Report No. 7

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DEVELOPMENT OF TREATMENTS PRODUCING LOW-FRICTION SURFACES ON ELASTOMERS

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TABLE OF CONTENTS

ABSTRACT

Our current objective, process optimization, appears to be nearing completion. The work done during this report period is leading to the optimization of two methods of treatment and treatment of samples for in-use or simulated in-use testing. Descriptions of the treatments of samples listed below are included in the report.

1) Water lubricated shaft bearings for U.S. Navy Engineering Experiment Station.

2) Retractable mast seals (chevron) for Portsmouth Naval Shipyard.

3) B138 and B142 experimental compounds from Puget Sound Naval Shipyard.

4) Four inch I.D. Buna N butterfly valve liners.

SAMPLE TREATMENT

During this period, samples were treated for three separate sources within the Navy and one industrial source with direct naval application. These samples have been returned to their sources and we are awaiting the return of test data.

Two sets of samples were received from the Puget Sound Naval Shipyard. One set consisted of four "O" rings; the other, tensile slabs and compression-set buttons of new compound formulations.

The "O" rings were identical in size to the "O" rings described in previous reports. No information on these rings was received except that they conformed to the specifications listed in Mil P5516. The treatment given these samples is described in Experiment 264. All were treated on the steel mandril constructed for a previous set of samples. All were slippery on the outer half and virtually unchanged on the inner half. These samples were returned via Code 634C.

The second set of samples consisted of tensile slabs and compression-set buttons fabricated from two special compounds developed at the Puget Sound Naval Shipyard. These compounds were identified by the numbers B138 and B142. Our objective was to find a satisfactory treatment for each of the compounds; treat and return any available tensile slabs and all the compression-set buttons. Initial treatments were done on tensile slabs; frictional and physical tests were run (see Exp. 236). Good coefficients of friction were

obtained by the various treatments, and it should be noted that although several samples ran extremely hot, they did not seize. The frictional surfaces also became extremely hard due to post cure effects. No significant loss of tensile properties was evidenced by comparison with the retain samples. The retains, however, exhibited much lower physical properties than were originally recorded. Puget Sound Naval Shipyard was notified by a letter report of our findings. Because of the extreme loss of properties on room-temperature storage, we do not plan any further work on these compounds unless specifically requested to do so.

During this period in Experiment 234, we treated four B[illegible] Butterfly valve liners for the [illegible] Rockwell Co., of Fairfield, Connecticut. Mr. A. Zellc, Director of Engineering, informs us that these liners will be tested and the results reported to us as soon as previously started tests are completed. This data should be available by the end of the next report period.

A visit was made to the Portsmouth Naval Shipyard, Kittery, Maine to explain the possible advantages of the "Slippery Rubber" process in their particular applications. As a result we obtained a set of chevron seals currently being tested for retractable mast applications. These seals were highly filled elastomer, typified by the AN6[illegible]23-40 specification. These samples were treated in Experiment 262. The effect of the treatment was to produce a very slippery surface. Tensile properties, however, were severely reduced. Since there is no flexing required in the application

it was deemed of sufficient interest to warrant testing. These tests will be run by the Portsmouth Naval Shipyard and reported to us via Code 634 when completed.

A set of six water-lubricated shaft bearings were received from the U. S. Navy Engineering Experiment Station at Annapolis, Maryland. These samples are prototypes of the external propeller shaft bearings used on large ships. They are fabricated from Buna N elastomer bonded to brass supporting plates. These specimens were treated in Experiment 230. A reduction of at least two-thirds in the low-speed unlubricated C_f was obtained. These samples were returned to the U. S. Navy Engineering Experiment Station for simulated in-use testing. The results of these tests will be forwarded to us when they are completed.

FLUORINATION PROCEDURE

During this report period the high-speed wear tests on Experiments 210, 216, 222 and 224 have been approximately two-thirds completed. These test results will be found at the beginning of the experimental portion of this report. The results completed to date indicate the following:

a. That the use of the BF_3-SF_4 complex produces a more slippery surface. The increased slipperiness is, however, offset by severe hardening of the surface which produces cracking on flexure.

2. That increased temperature in the fluorination procedure produces an increase in wear life;

3. That the time of fluorination is not directly related to the temperature of fluorination, but is also a function of the permeability of the elastomer itself and/or the previous history of the compound.

The lack of reproducibility of apparently duplicate samples remains inexplicable. Two possible solutions remain. The first, poor circulation of the fluorinating medium, will be eliminated by the installation of a magnetically driven fan in the pressure container. The second must be a function of the base elastomer itself. If the compounding ingredients are not homogeneously dispersed in the rubber matrix, this can be resolved by master batching a compound in the normal manner and then milling portions for longer periods of time. By this method it should be possible to determine whether or not the specific composition of small areas of the test specimen contributes significantly to the wear life.

A series of 15 runs, Experiment 246, evaluating the effect of time, temperature, $BF_3 \cdot SF_4$ complex concentration and SF_4 concentration of samples having no treatment other than cleaning and drying has been made. All data except high speed wear testing are tabulated in the experimental section of this report. Our initial evaluation of this series is reported in the following section of the report.

CONCLUSIONS

Our tentative conclusions that the entire treatment could be accomplished within the pressure vessel have been borne out by the results of Experiment 226. Wear lives of 2000 hours for Buna N, 52 hours for Neoprene, 2000 hours for Natural rubber and 166 hours for SBR were obtained. These samples treated for one hour at 115°C under autogeneous pressure show the feasibility of this time-saving method. A more complete experiment, No. 246, covering three temperatures, SF_4 and BF_3-SF_4 complex loadings, and three time-temperature levels has been partially evaluated. Complete evaluation of these results should indicate the proper treatment to use for first evaluation of samples received for the slippery rubber treatment.

We now have two graft-fluorination methods available for treating samples. Both of these methods involve the use of an unfluorinated monomer as one of the starting materials and SF_4 to produce the final slippery surface. Neither of these methods will be satisfactory for all samples but by using either one, or a combination of both, we have been able to successfully slipperize all samples which have thus far been submitted. We are looking forward to receiving more samples for treatment and subsequent testing.

FUTURE WORK

During the coming period we will complete the wear tests on the one-step graft fluorination procedure and investigate the possibility of using sulfur dichloride and sodium fluoride to

accomplish the fluorination of samples in the low-pressure reaction vessel. In addition, we plan to prepare low molecular weight oligomers having the basic chain unit of $\left(CH_2\underset{CF_3}{\underset{|}{CH}}- \right)$. It will be necessary to prepare these oligomers by indirect methods since low molecular weight polyacrylic acid polymers are inaccessible by direct methods. Methods for grafting there oligomers to base elastomers will be developed and the resultant surfaces subjected to the same test program as currently produced samples.

WEAR-LIFE DATA ON PREVIOUSLY REPORTED EXPERIMENTS

Experiment 210

Sample No.	Min. C_f	Max T^oF	Hours
2a	[illegible]18	18[illegible]	200+
2b	[illegible]	[illegible]	[illegible]
[illegible]a	[illegible]8	28[illegible]	200[illegible]

Experiment 216

Sample No.	Min. C_f	Max T^oF	Hours
[illegible]	.1[illegible]	[illegible]	[illegible]
9	[illegible]	[illegible]	[illegible]
10	[illegible]	[illegible]	[illegible]
[illegible]2	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]

Experiment [illegible]

Sample No.	Min. C_f	Max T^oF	Hours
[illegible]	[illegible]	[illegible]40	4[illegible]
[illegible]	[illegible]	[illegible]	[illegible]

Experiment [illegible]

Sample No.	Min. C_f	Max T^oF	Hours
[illegible]8	[illegible]	[illegible]	[illegible]00[illegible]
[illegible]2	.2[illegible]	[illegible]5	[illegible]

EXPERIMENTAL

Experiment No. 230 - Treatment of Bearings for U. S. Navy Engineering Experiment Station, Annapolis, Maryland

All Samples

20 min. @ 55-60°C in 100% Acrylic acid

30 min. U.V.

Fluorination

Time @ temperature	60 min.
Average temperature °C	115
psig maximum	12.25
SF_4 used	20.0 g

C_f @ in./sec	1.21	2.4
1	.620	.58[illegible]
2	.613	.600
3	.555	.52[illegible]
4	.54[illegible]	.533
5	.46[illegible]	.580
6	.647	.647

Samples returned to USNEES for testing

Experiment No. 232

Check samples for reproducibility

All samples Buna N

All samples 20 min @ 60°C in 100% Acrylic Acid

All samples 30 min U.V.

Fluorination

Sample No	1,2	3
Time @ Temp.	60 min	60
Average T°C	115°	115°
Maximum T°C	117.2	120
Maximum psig	18.0	12.25
SF_4 used	30.7	8.2
BF_3 [illegible] used	0	23.0

	Thickness Final	Shore A Initial	Shore A Final	C_f @ in/sec .21	C_f @ in/sec 2.4	C_f @ in/sec 120	Min C_f	Wear Life Max T°F	Wear Life Hrs.	Surface Condition
1	.126	45	48	.400	.423	.19[illegible]	.184	265	200+	
[illegible]	.126	4[illegible]	48	.400	.435	.199	.184	380	200+	
[illegible]	[illegible]	[illegible]5	[illegible]	[illegible]9	.[illegible]6[illegible]	.184				
[illegible]	[illegible]	[illegible]	53	[illegible]81	.470	[illegible]9				S1 H

	Modulus 100%	Modulus 200%	Modulus 300%	Tensile % Elong	Tensile psi	Comp. Set
[illegible]	16[illegible]	[illegible]90	450	590	[illegible]20	
[illegible]	[illegible]	[illegible]80	430	[illegible]80	900	
[illegible]	[illegible]	4[illegible]	606	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	590	[illegible]50	[illegible]9[illegible]

Experiment No. 234

Sample 1 No pretreatment - Fluorinated with Exp. 232 - very hard

Sample 2 No pretreatment - Fluorinated with Exp. 230

Sample 3 20 min. Acrylic at room temperature

16 min. U.V., no cracking

Treatment of complete liner

Time at temperature	2 hrs.
Average temperature °C	115
Maximum temperature °C	[illegible]7.2
psig maximum	[illegible]2.[illegible]
SF_4 used	[illegible].[illegible] g

Samples insufficiently slippery

Sample 1 retreated Exp. [illegible]6 - slippery

Sample 2 and 3 - 1[illegible] min @ 60° in [illegible]% Acrylic acid - [illegible] U.V.

Refluorinated

Time @ Temperature °C	[illegible] hrs
Average temperature °C	[illegible]
Maximum temperature	[illegible]
psig maximum	[illegible]7.[illegible]5
SF_4 used	28.3

Samples returned [illegible] for testing

Experiment No. 236 - Treatment of Compounds B138 and 142 for P.S.N.S.

1 - B138 30 min Acrylic acid @ 60°C - 30 min. U.V.

2 B142 30 min. Acrylic acid @ 60°C - 45 min U.V.

3 B138 3 min Acrylic acid @ 60°C - 45 min U.V.

4 B142 3 min Acrylic acid @ 60°C - 45 min. U.V.

5 B138 3 min. Acrylic acid @ 60°C - 60 min U.V.

6 B142 3 min. Acrylic acid @ 60°C - 60 min. U.V.

7 B138 45 min. Acrylic acid - 40 min. U.V.

8 B142 30 min. Acrylic acid - 40 min. U.V.

9 B138 No pretreatment

10 B142 No pretreatment

Further work stopped because of loss of tensile on room temperature storage.

Sample No.	Shore A Initial	Shore A Final	C_f @ in/sec. 1.21	C_f @ in/sec. 2.4	C_f @ in/sec. 120	Wear Life Min C_f	Wear Life Max T°F	Wear Life Hrs.	Surface Condition
1			.3[illegible]7	.354	.225	.135	460	69*	soft
2			.369	.374	.160	.168	510	48½*	soft
3			.4[illegible]	.438	.143	.143	200	[illegible]	soft
4			.362	.382	.184	.168	525	72*	soft
5			.354	.369	.152	.135	195	.1	soft
6			.385	.400	.191	.168	210	.1	soft
7	[illegible]	[illegible]9	.[illegible]50	.39[illegible]	.096	.116	310	100	soft
8	[illegible]	69	.418	.486	.219	.096	270	87	soft
9									soft
10									soft

* Test terminated because of high temperature

Experiment 236 (Continued)

Sample No.	Modulus 100%	200%	300%	Tensile % Elong.	psi	Comp. Set
7	310	602	1060	460	1840	60.4
8	830			150	1650	35.4

Experiment No. 240 - Preparation of $BF_3 \cdot SF_4$ complex for Experiment 242

Method of preparation - as previous

Yield 303.8 g.

Experiment No. 242 - Expansion of Experiment 174

20 min. @ 60°C - 100% Acrylic acid

No U.V.

Thickness Final	Shore A Int.	Shore A Final	C_f @ in/sec. 1.21	2.4	120	Wear Min C_f	Max. T	Hr	Surface Condition
[illegible]	52	49	.606	.731	45	--	[illegible]	nil	soft

Modulus 100%	200%	300%	Tensile % Elong.	psi	Compression Set
15[illegible]	304	496	587	1139	50.0

Experiment No. 244 - Expansion of Experiment [illegible]4 - Development of one-step graft fluorination method

Time @ temperature	60 min.
Average temperature °C	[illegible]
psig maximum	1735
SF_4 used	35.0
Acrylic acid used	15.0

All samples had a tacky surface film which dissolved off with an acetone rinse.

Experiment No. 244 (Continued)

Sample	Thickness Final	Shore A Init	Shore A Final	C_f @ in/sec. 1.21	2.4	120	Wear Min. C_f	Max T^oF	Hrs.
Buna	124	49	49	.456	.582	.219	.213	280	200*
Neoprene	133	57	61	.672	.759	.239	.232	475	52*
Natural	138	60	64	.513	.581	.160	.152	225	214
SBR	142	62	65	.657	.605	.176	.063	170	166

* Test terminated because of high temperature

Sample	Modulus 100%	200%	300%	Tensile % Elong.	psi	Compression Set
Buna	177	295	470	477	775	95.5
Neoprene	358	558	840	[illegible]70	1080	94.9
Natural	378	983	[illegible]744	343	2047	70.7
SBR	451	1[illegible]85	1974	31[illegible]	2145	69.5

Experiment No. 246 - Series of 13 experiments based on Experiment [illegible]74 to determine optimum treatment for combined graft-fluorination treatment in the low pressure reaction vessel.

One sample each of Buna N, Neoprene, Natural, SBR and Butyl in each run.

Bomb Run No.	1	2	[illegible]	4	5	6	[illegible]	8	9	10	11	12	13
Av. Temp. °C	145	145	145	145	145	13[illegible]	11[illegible]	[illegible]5	[illegible]5	1[illegible]5	[illegible]5	115	145
Time @ T	80	80	40	40	6[illegible]	6[illegible]	6[illegible]	40	80	40	8[illegible]	40	60
Max. T°	147	147	[illegible]47	147	148.5	136.5	[illegible]22.[illegible]	[illegible]25.5	124.[illegible]	[illegible]26.8	[illegible]26.8	[illegible]25.5	148.4
psig max.	24.0	14.25	28.[illegible]	12.7	19.25	18.2	19.0	5.7*	[illegible]3.[illegible]	20.0	28.[illegible]	[illegible]6.1	[illegible]4.8
SF_4	[illegible]5	[illegible]5	45	1[illegible]	[illegible]2.8	[illegible]0.0	30.0	6.[illegible]	6.[illegible]	[illegible]2.8	[illegible]9.2	19.2	[illegible]2.8
$BF_3 \cdot SF_4$	0	0	0	0	27.0	[illegible]	0	[illegible]4.0	1[illegible].[illegible]	28.0	42.0	42.0	28.0
Ratio BF_3/SF_4	0	0	0	0	.35[illegible]	0	0	.36	.36	.36	.35	.36	.36

*Pressure leak in bomb

BUNA N

Bomb run Number	Thickness Initial	Thickness Final	Shore A Initial	Shore A Final	C_f @ in/sec. 1.21	C_f @ in/sec. 2.4	C_f @ in/sec. 120
1	.122	.123	55	58	.50	.54	>.46
2	.124	.124	70	73	.71	.79	>.46
3	.124	.124	70	76	.44	.50	.305
4	.125	.124	70	70	.96	.94	>.46
5	.124	.124	71	76	.27	.31	.168
6	.124	.124	71	72	.61	.67	.346
7	.124	.125	70	72	.71	.73	.346
8	.125	.125	70	69	.285	.350	.199
9	.124	.124	70	72	.30	.34	.184
10	.124	.124	71	76	.33	.39	.199
11	.124	.124	70	80	.32	.36	.152
12	.123	.124	70	76	.35	.36	.176
13	.125	.125	70	80	.33	.37	>.46

BUNA N

Bomb Run Number	Modulus 100%	200%	300%	Tensile % Elong.	psi	Compression set
1	260	580		220	720	37.8
2	910			170	1760	33.6
3	1310			103		38.3
4	820	1390		220	2010	32.7
5	830			150	1210	43.0
6	970			160	1530	36.1
7	880	1720		200	1720	35.8
8	720	1500		220	1610	44.0
9	810			190	1465	39.2
10	860	1530		200	1520	44.1
11	820			140	1000	49.9
12	870			170	1310	46.4
13	760			130	915	45.9

NEOPRENE

Bomb Run Number	Thickness Initial	Thickness Final	Shore A Initial	Shore A Final	C_f @ in/sec. 1.21	C_f @ in/sec. 2.4	C_f @ in/sec. 120
1	.133	.136	60	68	.60	.62	.324
2	.131	.131	60	66	.73	.86	>.46
3	.132	.134	60	67	.69	.62	.324
4	.133	.133	61	64	.75	.99	>.46
5	.134	.135	61	68	.37	.43	.29
6	.131	.133	61	65	.51	.65	>.46
7	.131	.133	61	66	.56	.69	>.46
8	.133	.133	62	62	.44	.54	.279
9	.133	.134	64	67	.35	.41	.238
10	.133	.134	62	67	.46	.55	.268
11	.133	.150	60	70	.42	.48	.295
12	.137	.140	60	71	.42	.53	.232
13	.127	.131	60	70	.42	.52	.239

NEOPRENE

Bomb Run Number	Modulus 100%	200%	300%	Tensile % Elong.	psi	Compression Set
1	390	630		280	810	71.2
2	350	540	810	310	860	70.9
3	300	430	660	320	720	78.7
4	280	400	640	370	850	77.6
5	320	490	720	370	920	69.4
6	300	440	680	360	880	83.8
7	290	425	680	390	960	83.2
8	320	530		390	1150	83.5
9	270	390	650	390	930	79.3
10	260	390	635	370	885	81.2
11	310	490	710	380	870	81.1
12	330	505	730	370	940	84.1
13	295	400	525	400	735	64.6

NATURAL

Bomb Run Number	Thickness Initial	Thickness Final	Shore A Initial	Shore A Final	C_f @ in/sec. 1.21	C_f @ in/sec. 2.4	C_f @ in/sec. 120
1	.137	.137	65	67	.92	.81	.324
2	.140	.140	64	65	.81	NR	.239
3	.141	.142	65	67	.62	.62	.279
4	.140	.140	65	64	NR	NR	>.46
5	.138	.139	65	70	.37	.42	.333
6	.139	.140	65	64	.42	.47	.428
7	.138	.140	64	64	.66	.75	.226
8	.139	.141	63	66	.35	.44	.29
9	.138	.137	65	68	.38	.47	.342
10	.137	.138	65	70	.39	.45	.359
11	.136	.139	65	72	.39	.47	.324
12	.137	.139	65	70	.37	.42	.274
13	.133	.135	65	70	.37	.46	.351

NATURAL

Bomb Run Number	Modulus 100%	200%	300%	Tensile % Elong.	psi	Compression Set
1	430	1060	1890	300	1910	53.3
2	415	1080	1945	360	2410	48.0
3	440	1030	2060	360	2610	52.9
4	430	1140	2070	380	2725	44.8
5	400	980	1690	350	2025	57.0
6	430	1190	2090	380	2780	55.3
7	410	1100	1990	400	2940	55.6
8	430	1020	1890	380	2450	62.0
9	470	1160	1970	360	2360	56.8
10	445	1120	1910	350	2220	62.3
11	435	1020	1830	350	2110	59.1
12	380	970	1750	400	2480	75.8
13	390	925	1600	360	2020	70.6

SBR

Bomb Run Number	Thickness Initial	Thickness Final	Shore A Initial	Shore A Final	C_f @ in/sec. 1.21	C_f @ in/sec. 2.4	C_f @ in/sec. 120
1	.140	.141	65	67	.63	.76	>.46
2	.142	.142	64	66	NR	NR	.219
3	.143	.143	65	66	.61	.56	.329
4	.140	.140	65	64	NR	NR	>.46
5	.144	.144	65	70	.40	.46	.279
6	.141	.142	65	64	.50	.59	.411
7	.140	.140	65	63	1.44	NR	.251
8	.140	.147	61	67	.35	.45	.300
9	.145	.146	66	69	.40	.48	.346
10	.142	.144	65	70	.39	.45	.285
11	.141	.144	65	70	.39	.49	.279
12	.133	.140	65	70	.37	.47	.274
13	.137	.140	65	70	.38	.46	.342

SBR

Bomb Run Number	Modulus 100%	Modulus 200%	Modulus 300%	Tensile % Elong.	Tensile psi	Compression set
1	455	1220		280	1960	55.9
2	400	1040	1920	350	2310	48.7
3	400	1150	2050	320	2330	56.3
4	380	985	1820	430	2850	50.4
5	430	980	1755	350	2080	55.0
6	450	1190	2130	370	2690	58.4
7	400	1070	1900	400	2675	55.2
8	400	1055	1860	400	2570	63.4
9	470	1150	2010	340	2280	61.5
10	480	1240	2080	350	2400	52.3
11	430	1070	1835	330	2050	62.9
12	410	1050	1900	350	2350	77.4
13	390	930	1600	360	2020	70.6

BUTYL

Bomb Run Number	Thickness Init.	Thickness Final	Shore A Init.	Shore A Final	C_f @ in/sec. 1.21	C_f @ in/sec. 2.4	C_f @ in/sec. 120	Surface Condition
1	.125	.125	52	53				soft
2	.125	.125	55	51				soft
3	.123	.123	55	52				soft
4	.123	.123	54	50				soft
5	.122	.125	52	50	.60	.65	.338	soft
6	.121	.122	53	49				soft
7	.123	.124	54	50				soft
8	.121	.122	50	48				soft
9	.126	.125	54	48				soft
10	.123	.121	54	51				soft
11	.124		52					s. tacky
12	.124	.128	55	52				s. tacky
13	.124	.136	54	50				s. tacky

BUTYL

Bomb Run Number	Modulus 100%	200%	300%	Tensile % Elong.	psi	Compression Set
1	340	840	1240	330	1350	69.3
2	240	630	1090	340	1230	63.1
3	310	780	1210	360	1375	71.9
4	300	750	1190	350	1320	71.4
5	300	750	1170	330	1280	
6	300	745	1181	400	1420	76.0
7	325	820	1220	390	1420	75.2

Experiment No. 262 - Treatment of Chevron seals for Portsmouth Naval Shipyard

Retractable Mast seals

Treated with one-step graft-fluorination method

Time @ temperature	60 min.
Average temperature	115°C
Maximum psig	11.3
SF_4 used	20.0
Acrylic Acid used	10.0 g.

Samples were slightly harder but with a sufficiently low coefficient of friction to warrant in application testing. Testing will be done at Portsmouth Naval Shipyard and results forwarded through Code 634C.

Experiment No. 264 - Evaluation of new rubber stocks

Samples of Buna N - SBR - Natural rubber

Fluorination by one-step graft fluorination method

Time @ temperature	60 min.
Average temperature	115^{o}C
Maximum psig	16.2
SF_4 used	30.0 g.
Acrylic acid used	13.0 g.

All samples have satisfactory appearance and are comparable to previous stocks.

Experiment No. 266 - "O" Rings (Mil P 5516) for Puget Sound Naval Shipyard

All rings treated as previous on outside surface only

Fluorination by one-step graft-fluorination method with Experiment 264

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